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IS 8400 (1976) : 2, 4-dinitro Chlorobenzene [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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“Knowledge is such a treasure which cannot be stolen”



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IS : 8400 - 1976

Indian Standard
SPECIFICATION FOR
2, 4-DINITRO CHLOROBENZENE

UDC 667.285.1 : 547.548



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1977

AMENDMENT NO. 1 MAY 2002
TO
IS 8400 : 1976 SPECIFICATION FOR 2,4-DINITRO
CHLOROBENZENE

(*Page 3, clause 0.3, Structural Formula*) — Insert the following below the structural formula:

'(CAS No. 97 00-7)'.

[*Page 4, Table 1, Sl No. (iii)*] — Insert the following after Sl No. (iii):

(1)	(2)	(3)	(4)
iv) Matter insoluble in methanol, percent by mass, <i>Max</i>	0.2	when tested as per	IS 5299 : 2001

(PCD 11)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR

2, 4-DINITRO CHLOROBENZENE

Dye Intermediates Sectional Committee, CDC 46

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(Continued on page 2)

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IS : 8400 - 1976

(Continued from page 1)

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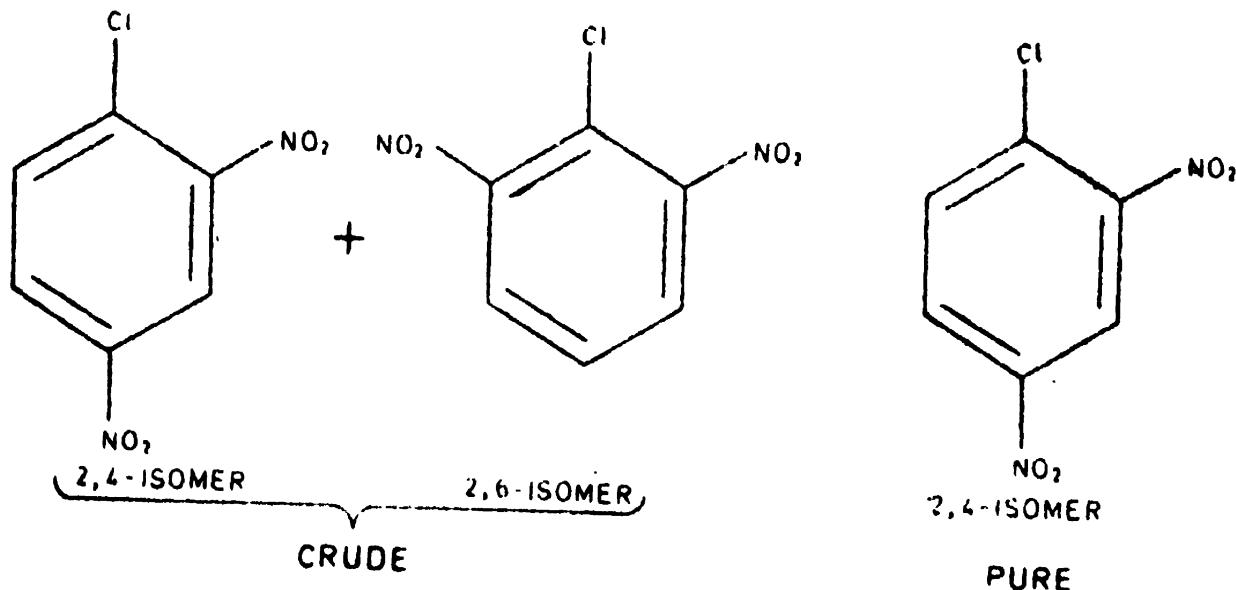
Indian Standard
SPECIFICATION FOR
2, 4-DINITRO CHLOROBENZENE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 August 1976, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 2,4-Dinitro chlorobenzene ($C_6H_3O_4N_2Cl$) is an important dye intermediate. It is supplied in two grades, namely, crude and pure.

0.3 Crude dinitro chlorobenzene is generally a mixture of 2,4- and 2,6-isomers (95 percent 2,4-isomer and 5 percent 2,6-isomer), whereas pure dinitro chlorobenzene is 2,4-isomer. These are represented by the following structural formulae:



**DINITRO CHLOROBENZENE
(MOLECULAR MASS 202.5)**

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 2,4-dinitro chlorobenzene.

2. GRADES

2.1 There shall be two grades of the material, namely:

- a) *Grade 1* — Pure, and
- b) *Grade 2* — Crude.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of crystalline mass.

3.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 2, 4-DINITRO CHLOROBENZENE

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST REF TO CL NO. IN APPENDIX A
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Crystallizing point, °C, Min	49·5	47·0	A-1
ii)	Assay, percent by mass, Min	99·5	95	A-2
iii)	2, 6-Dinitro chloro- benzene, percent by mass, Max	—	5	A-2

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in steel drums (*see IS : 2552-1970**) or as agreed to between the purchaser and the supplier. The container shall be securely closed.

4.2 Marking — Each container shall bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number;
- d) Tare, net mass and gross mass; and
- e) The minimum cautionary notice worded as under:

‘BLOOD POISON. IRRITATES SKIN. INHALATION
OF DUST MAY BE DETRIMENTAL TO HEALTH.’

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE—The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

5.2 Number of Tests—Tests for crystallizing point, assay and 2, 6-dinitro chlorobenzene shall be conducted on each of the individual samples.

5.3 Criteria for Conformity—The lot shall be declared as conforming to the requirements of crystallizing point, assay and 2, 6-dinitro chlorobenzene if each of the results obtained as in 5.2 satisfy the corresponding requirements given in Table 1.

6. TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 5 of Table 1.

6.2 Quality of Reagents—Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE—‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table 1, and Clause 6.1*)

METHODS OF TEST FOR 2, 4-DINITRO CHLOROBENZENE

A-1. CRYSTALLIZING POINT

A-1.1 Determine the crystallizing point of the material as prescribed in 7 of IS : 5299-1969*.

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (second revision).

A-2. ASSAY AND 2, 6-DINITRO CHLOROBENZENE

A-2.0 Outline of the Method — The contents of 2,4-and 2,6-dinitro chlorobenzenes are determined by gas chromatographic analysis.

A-2.1 Apparatus

A-2.1.1 Gas Chromatograph — with flame-ionization detector.

A-2.1.2 Potentiometric Strip-Chart Recorder — full scale deflection 1 second.

A-2.1.3 Temperature Programmer

A-2.1.4 Column — made of glass, 2 m long, 6 mm in diameter, and packed with 10 percent Apizon L on 72/85 mesh Celite* or an equivalent material.

A-2.2 Reagents

A-2.2.1 2, 6-Dinitrotoluene — internal standard.

A-2.2.2 2, 4-Dinitro chlorobenzene

A-2.2.3 2, 6-Dinitro chlorobenzene

A-2.2.4 Acetone — solvent.

A-2.3 Procedure

A-2.3.1 Operating Parameters of Gas Chromatograph

A-2.3.1.1 Injection port temperature — 275°C.

A-2.3.1.2 Column temperature — 165°C.

A-2.3.1.3 Carrier gas — Nitrogen, 45 ml/min.

A-2.3.1.4 Chart speed — 25 cm/h.

A-2.3.2 Sample — Dissolve 10 g of the sample in acetone. Add a known mass of the internal standard and make up the volume to 100 ml with acetone. Inject 0·4 microlitre.

A-2.3.3 Calibration Factor — Internal standardization is the method used for calibration of the results of analysis. Make up and chromatograph known mixtures of component 'n' for which the calibration factor is to be determined together with the internal standard plus other components of the sample. Calculate the calibration factor as follows:

$$K_n = \frac{X_n \times A_s}{X_s \times A_n}$$

*These are imported chemicals and are generally available with the fabricators of gas chromatographs and also with the stockists of chemicals.

where

K_n = calibration factor for component 'n' against internal standard 's',

X_n = mass percent of component 'n' in the sample,

A_s = peak area for internal standard 's',

X_s = mass percent of internal standard 's' in sample, and

A_n = peak area for component 'n'.

A-2.3.4 Interpretation of Chromatogram — Elution order of components is as follows:

- Acetone (solvent used);
- Mononitro chlorobenzene, if any;
- 2, 6-dinitrotoluene (internal standard);
- 2, 6-dinitro chlorobenzene; and
- 2, 4-dinitro chlorobenzene.

A-2.4 Calculation

$$\text{Component 'n' in the sample, percent by mass} = \frac{K_n \times A_n \times X_s}{A_s}$$

where

K_n = calibration factor for component 'n' (see A-2.3.3),

A_n = peak area for component 'n',

X_s = mass percent of internal standard added to the sample, and

A_s = peak area for internal standard 's'.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>	<i>Conversion</i>
Force	newton	N	1 N = 1 kg·1 m/s ²
Energy	joule	J	1 J = 1 N·m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V·s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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